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Burner Rig Evaluation of Thermal Barrier Coating Systems for Nickel-Base Alloys

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COATING SYSTEMS FOR NICKEL-BASE ALLOYS

Michael A. Gedwill

ABSTRACT

Eight plasma-sprayed bond coatings were evaluated for their potential use with ZrO_2 - Y_2O_3 thermal barrier coatings (TBCs) which are being developed for coal-derived-fuel-fired gas turbines. Longer TBC lives in cyclic burner rig oxidation to 1050° C were achieved with the more oxidation resistant bond coatings. These were Ni-14.1Cr-13.4Al-0.10Zr, Ni-14.1Cr-14.4Al-0.16Y, and Ni-15.8Cr-12.8Al-0.36Y on René 41. The TBC systems performed best when 0.015-cm thick bond coatings were employed that were sprayed at 20 kW using argon - 3.5 v/o hydrogen. Cycling had a more life limiting influence on the TBC than accumulated time at 1050° C.

BURNER RIG EVALUATION OF THERMAL BARRIER COATING SYSTEMS FOR NICKEL-BASE ALLOYS

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SUMMARY

Eight NiCrAl bond coatings were evaluated for their potential to improve the durability of ceramic thermal barrier coatings (TBC) on nickel-base alloys for use in gas turbines. A Mach 0.3 burner rig fired with Jet A fuel was used to rate the cyclic performance of a 0.038-cm thick $ZrO_2-12Y_2O_3$ TBC with various bond coatings on solid René 41 pins at a metal temperature of 1050° C. The effects of bond coating thickness, plasma spraying conditions, and frequency of thermal cycling were also evaluated. TBC performance was rated according to its resistance to macroscopic cracking. The mode of TBC failure and the extent of bond coating degradation were examined by post-test metallography.

Oxidation resistance of the bond coatings strongly influenced the performance of the TBC. Longer TBC lives were achieved with the more oxidation resistant bond coatings. The best bond coatings were Ni-14.1Cr-13.4Al-0.10Zr, Ni-14.1Cr-14.4Al-0.16Y, and Ni-15.8Cr-12.8Al-0.36Y. TBC performance was sensitive to the Cr and Y contents with low-Cr-containing coatings (14 to 18 w/o) giving better performance; the optimum Y content appeared to be 0.3 w/o.

The TBC systems performed best when 0.015-cm thick bond coatings were employed that had been sprayed at 20 kW using argon - 3.5 w/o hydrogen. Either decreasing the thickness to 0.010 cm or deviating from the 20 kW power level resulted in inferior coating system performance.

Cycling was found to have a more life-limiting influence on the TBC than accumulated time at elevated temperature. The TBC lives, in terms of time at temperature, increased markedly when 1.0-hour rather than 0.1-hour heating cycles were used with the better bond coatings, very little oxidative degradation of the bond coating occurred, and TBC life appeared to be limited by macrocrack initiation and growth in the ceramic coating induced by thermal cycling.

INTRODUCTION

With ceramic thermal barrier coatings (TBC) applied to the outer surfaces of cooled airfoils, gas turbines can be designed to obtain improved efficiency and/or durability. These coatings are being actively developed for clean-fuel-fired aircraft engines and for dirty-fuel-fired industrial/utility engines (refs. 1 to 13).

The durability of TBCs is being continually improved through the use of better ceramic and bond coatings. Although various TBC systems are being studied, the most interest is in a plasma-deposited, two-layer system consisting of a 0.013 to 0.064-cm thick $ZrO_2-Y_2O_3$ top coat, and a 0.005- to 0.020-cm thick NiCrAlY bond coating.

In studies involving cyclic furnace, torch, and burner rig tests, it was shown that the durability of TBCs is strongly influenced by the oxidation resistance of the bond coatings (refs. 8 and 14). In cyclic furnace-test data reported in reference 14 by the present author, it was found that the

1100° C oxidation resistance of bond coatings alone, deposited on the nickel- and cobalt-base alloys, B-1900+Hf and MAR-M509, is greatly dependent on plasma spraying conditions and thickness. Also, sufficient surface roughness coupled with good oxidation resistance of the bond coatings are required for improved adherence of a $\text{ZrO}_2\text{-12Y}_2\text{O}_3$ TBC (all compositions expressed in weight percent, unless otherwise noted). In that study, the most oxidation-resistant bond coatings on B-1900+Hf were Ni-14.1Cr-13.4Al-0.10Zr, Ni-14.3Cr-14.4Al-0.16Y, and Ni-15.8Cr-12.8Al-0.36Y. In a recent study involving Mach 0.3 burner rig tests at 1040° C metal temperature (ref. 13), it was reported that the lives of TBCs ($\text{ZrO}_2\text{-8Y}_2\text{O}_3$ and $\text{ZrO}_2\text{-12Y}_2\text{O}_3$) on solid, nickel-base alloy René 41 pins were longer with a Ni-18Cr-12Al-0.3Y bond coating than with Ni-16Cr-6Al-0.3Y. Some Mach 0.3 burner rig tests were also conducted on air-cooled erosion bars using simulated utility fuels (refs. 10 and 12). In these tests, Jet A combustion gases doped to the fuel equivalent of 5 ppm Na and 2 ppm V were used; the metal substrate temperature was 840° C, and the substrate was nickel-base alloy IN-792. In these tests, the lives of TBCs ($\text{ZrO}_2\text{-8Y}_2\text{O}_3$ and Ca_2SiO_4) with a Ni-15.8Cr-12.8Al-0.36Y bond coating were at least doubled compared to those with the less oxidation resistant Ni-16Cr-6Al-0.31Y bond coating.

The purpose of the present study was: first, to further evaluate the three bond coatings identified in the furnace tests of reference 14 along with three new ones that appear to have the potential to improve the durability of TBCs on nickel-base alloy airfoils for use in gas turbines; and second, to recommend two bond coatings for future evaluation on air-cooled erosion bars in a Mach 0.3 burner rig fired with a "dirty fuel". For comparison, two bond coatings with slightly less oxidation resistance (Ni-17.7Cr-12.2Al-0.11Y and Ni-30.9Cr-11.1Al-0.48Y) were included in the study (ref. 14). A Mach 0.3 burner rig fired with Jet A fuel was used to rate the performance of a $\text{ZrO}_2\text{-12Y}_2\text{O}_3$ TBC with the eight bond coatings on solid René 41 pins at a metal temperature of 1050° C. A limited investigation was made on the effects of bond coating thickness, plasma spraying conditions, and cycling frequency. The first two variables were reinvestigated to assure that they had the same effects on TBC life in the burner rig as they did in the cyclic furnace tests (ref. 14). TBC performance was rated by its resistance to macroscopic cracking. In addition, post-test metallographic analyses were performed to examine the extent of bond coating degradation and the mode of TBC failure.

MATERIALS, APPARATUS, AND PROCEDURES

Solid wrought René 41 (Ni-19Cr-11Co-10Mo-3Ti-1.5Al-0.09C-0.005B), 1.27-cm diameter pins were plasma spray coated with various bond coatings and $\text{ZrO}_2\text{-12Y}_2\text{O}_3$. The compositions of the plasma spray powders (all -200 + 325 mesh) are listed in table I. The procedures and parameters used for specimen preparation and plasma spraying are given in references 5 and 14. During coating, the pins were rotated at 200 rpm. The coatings were manually applied in air to target thicknesses listed in table II for the bond coatings and 0.038 cm, for the TBC. Each coating thickness was determined from measurements taken before and after coating. The bond coating thickness measurements are listed in table II. The measured TBC thickness values varied from 0.034 to 0.042 cm.

The Mach 0.3 burner rig employed in these tests is shown in figure 1 and has been fully described in reference 15. Briefly, eight specimens were

placed in a holder, which was rotated at 630 rpm in front of the burner nozzle. In these tests, the specimens were heated either for 6 minutes followed by a 3-minute air blast (Mach 0.7) quench, or for 60 minutes followed by the 3-minute air quench. The burner rig was fired at a fuel-to-air ratio of about 0.049 using Jet A fuel. A calibration test was made using a thermocouple embedded in the center of a coated specimen and the "test temperature" was determined to be $1050^{\circ} \pm 20^{\circ} \text{C}$. About 3 minutes in the flame was required for the specimens to reach the 1050°C "test temperature". At the end of the 3-minute air quench, the center of the specimen was at about 80°C . The maximum temperature in the hot zone on the front surfaces of the coated specimens, as measured with a disappearing filament optical pyrometer, was 1040°C . Since all of the TBC failures occurred or apparently started on the back surface of the specimens, an attempt was made to measure the maximum temperature in the hot zone on the back surface. A test run was made with a specimen having a thermocouple spot welded in a groove on the back surface before coating. The maximum (in heating) and minimum (in cooling) temperatures recorded with this specimen were 1153° and 127°C , respectively.

In these tests, the specimens were visually examined at least once every day. Specimens were tested until TBC failure (cracking and/or spalling of the ceramic coating). All specimens were run in duplicate. After test, the specimens were photographed, sectioned very slowly through the hot zone, metallographically prepared, and examined.

RESULTS AND DISCUSSION

The results obtained for the $\text{ZrO}_2\text{-}12\text{Y}_2\text{O}_3$ TBC with eight bond coating composition variations will be presented and discussed first. This will be followed by the effects of bond coating thickness and plasma spray conditions, and then by the effect of cycling frequency on TBC performance with two of the three best bond coatings. The results are summarized in table II.

A. Bond Coating Compositions

The effects of the eight bond coating composition variations on TBC performance in the burner rig test using 0.1-hour heating cycles are shown in figure 2. Based on the furnace-test data obtained in reference 14, the bond coatings were plasma sprayed at 20 kW using argon - 3.5 v/o hydrogen arc gas to a nominal thickness of 0.015 cm. The results of figure 2 suggest that the bond coatings can be classified into the following three groups relative to their effectiveness in improving the durability of the TBC in the burner rig test:

- Group I (most effective)
 - Ni-14.1Cr-13.4Al-0.10Zr
 - Ni-14.3Cr-14.4Al-0.16Y
 - Ni-15.8Cr-12.8Al-0.36Y
- Group II
 - Ni-17.7Cr-12.2Al-0.11Y
 - Ni-30.7Cr-12.0Al-0.25Y
- Group III (least effective)
 - Ni-28.8Cr-19.9Al-0.59Y
 - Ni-30.9Cr-11.1Al-0.48Y
 - Ni-38.6Cr-10.4Al-0.69Y

Within each group the coatings appeared to be about equally effective. The TBC life with any one of the Group I bond coatings was at least 2500 cycles in the burner rig test. Those in Group I are the same ones that were identified as the most oxidation resistant coatings on nickel-base alloy B-1900+Hf in cyclic furnace tests (ref. 14). The other two reference 14 bond coatings of lower oxidation resistance were Ni-30.7Cr-12.0Al-0.25Y (Group II) and Ni-30.9Cr-11.1Al-0.48Y (Group III). These observations confirm that the oxidation resistance of the bond coatings plays an important role on the performance of a TBC. Also, simple furnace oxidation testing of bond coatings alone deposited on the substrate is a good, first approach in evaluating bond coatings. For "dirty-fuel" fired environments, it is felt that additional evaluations should involve the TBC and bond coatings together, since TBC failure in these environments is attributed to condensation, penetration, and possible reaction(s) of the fuel impurities within the outer ceramic coating (refs. 11 and 12). However, even in dirty-fuel fired environments bond coating composition does strongly influence TBC life (ref. 10).

The results of figure 2 also indicate that the performance of the TBC was sensitive to the Y and Cr contents of the bond coatings. The performance of the TBC systems with the NiCrAl-0.36Y (Group I) and NiCrAl-0.25Y (Group II) coatings were much better in comparison to those with the NiCrAl-0.11Y (Group II) and NiCrAl-0.48Y (Group III) coatings, respectively. It would appear that the optimum Y content for a NiCrAlY bond coating should be about 0.3 weight percent in agreement with references 8 and 14. With respect to the Cr content, the TBC systems with the low Cr containing coatings (14 to 18 w/o) were better than those with the high Cr (28 to 39 w/o). However, the data of Stecura (ref. 8) indicate, in light of these results, that the optimum Cr level is influenced by the Al level since Ni-25Cr-6Al-0.3Y was the best bond coating in that study.

The TBC failures observed visually on the figure 2 specimens occurred by cracking followed by spalling of the ceramic coating (table II). Typical examples illustrating the extent of cracking and spalling of these TBC systems are presented in figure 3. All the failures of the ceramic coating apparently started on the back surface of the specimens (fig. 3(a)), which was at a higher temperature ($\sim 1150^\circ\text{C}$) in the burner flame than the front surface. (Maximum temperatures measured on metallic specimens in a burner flame were reported to be the highest on the back surface (ref. 16).) In some cases, the cracking/spalling progressed around the specimen to the front surface (fig. 3(b)), on further cycling if cracking was severe. The least effective, Group III bond coatings were also observed to have cracked and spalled at the end of testing (fig. 3(c)), except in one instance (table II). As is also evident in figures 3(a) and 3(b), the ceramic coating is discolored, primarily on the front surface. In general, all the specimens were discolored after about 1600 test cycles, i.e., after ~ 160 hours in the burner flame. The discoloration varied from reddish-brown to almost black. Examinations in a scanning electron microscope (SEM) using an energy dispersive X-ray spectrometer indicated that the discoloration was due to iron-containing deposits (probably iron oxide(s)). It is believed that the deposits were present in the combustion air because oily reddish-brown deposits have been observed in the combustion air flowmeters. It is believed that the deposits had no effect on the performance of these TBC specimens. The work of Zaplatynsky (ref. 17) showed that Fe_2O_3 does not react with $\text{ZrO}_2\text{-}8\text{Y}_2\text{O}_3$ at 1200°C for times up to 400 hours. The specimens tested in the present study using the 0.1-hour heat cycles were at $< 1150^\circ\text{C}$ for

less than 200 hours. Furthermore, the deposits were primarily on the front surfaces of the specimens while TBC failures (external cracking) started on the back surfaces.

Metallographic cross sections in the zone of maximum failure of two specimens after test are shown in figure 4. Figure 4(a) is typical of the tested specimens with the Group I bond coatings, while figure 4(b) is typical of those tested with the poorer Group III coatings. The ceramic coating on the front surfaces of the two specimens of figure 4 is in place, but cracked. Similar cracking within the ceramic near the ceramic/bond coating interface has been reported (refs. 7, 10, 14, and 18). On the back surfaces of these two specimens, the ceramic coating has spalled off, except for some small remnants attached to the bond coatings. With the best bond coatings (Group I), the ceramic coating failed well before the bond coatings were appreciably oxidized. The bond coating in figure 4(a), however, is less oxidized on the front and back surfaces after a longer exposure in the burner rig than the poorer bond coating shown in figure 4(b). Also, there is less oxide penetration into substrate in figure 4(a). The severe oxidation that occurred in the bond coating and along the substrate/bond coating interface on the back surface of the figure 4(b) specimen led to cracking and spalling of the bond coating similar to that shown previously (fig. 3(c)). The specimens with the Group II bond coatings (not shown in fig. 4) were oxidized to an intermediate degree. These observations suggest that volume expansion along the ceramic/metal interface as a result of bond coating oxidation plays a key role in unzipping the ceramic.

Based on performance in the burner-rig oxidation test and post-test evaluations, the Ni-14.1Cr-13.4Al-0.10Zr and Ni-15.8Cr-12.8Al-0.36Y bond coatings are the two recommended for future TBC evaluations on air-cooled, erosion bars in a burner rig fired with a "dirty fuel".

B. Bond Coating Thickness and Plasma Spraying Conditions

Figure 5 shows the effects of bond coating thickness and plasma spraying conditions on the performance of two TBC systems in the Mach 0.3 burner rig test using the 0.1-hour heating cycles. Increasing the thickness from 0.010 to 0.015 cm had a beneficial effect on performance, i.e., at least a 100 percent increase in the number of cycles to failure. In cyclic furnace testing of bond coatings deposited on nickel and cobalt-base alloys, it was shown that the oxidation resistance decreased drastically when the coating thickness was reduced from 0.015 to 0.010 cm (ref. 14). Although not investigated in the present study, bond coatings thicker than 0.015 cm might give further improvement in the burner rig lives of TBC systems. In a study involving cyclic furnace testing of various ZrO_2 - Y_2O_3 /NiCrAlY TBC systems, Stecura (ref. 8) concluded that increasing the average bond coating thickness to 0.020 cm was desirable. However, turbine choking may limit the benefits of thicker coating systems.

As shown in figure 5, the performance of TBC systems in the burner rig test was affected by the plasma arc power used in applying the bond coatings. Either decreasing or increasing the power from 20 kW resulted in poorer performance, i.e., about a 20- to 90-percent loss in the number of cycles to failure. These observations are in complete agreement with the performance of bond coatings deposited on nickel and cobalt-base alloys noted in the previous study using furnace testing (ref. 14). In that study, it was also found that a little hydrogen in the arc gas improved the oxidation resistance of the coatings. The results obtained in the burner rig and

furnace tests indicate that the TBC systems perform best when the bond coatings are plasma sprayed at 20 kW using Ar - 3.5 v/o H₂ and the conditions noted above in the Materials, Apparatus, and Procedures section. These spraying conditions, however, might not be the optimum conditions. A systematic parametric study of plasma spraying of both the bond coating and the ceramic coating would be desirable.

The TBC failures observed on the plasma-spray-condition specimens were similar to those noted above (i.e., cracking apparently starting on the back surface followed by spalling); examples were shown previously in figures 3(a) and 3(b).

Only a few plasma-spray-condition specimens were metallographically examined after testing. The metallographic observations noted on these specimens are in agreement with those reported above and in reference 14. That is, the oxidation resistance of the bond coatings is strongly affected by the plasma spraying conditions. Through their effect on bond coating oxidation resistance, bond coating plasma spraying parameters strongly influence TBC life.

C. Thermal Cycling Frequency

The effect of thermal cycling frequency on TBC performance is shown in figure 6 for two systems in the Mach 0.3 burner rig. Using the short, 0.1-hour heating cycles, the lives of the TBCs in the 1050° C rig test were less than 200 hours. The lives, however, were increased by a factor of about 2 to 5 with the longer, 1.0-hour heating cycles. These results, therefore, show that less frequent thermal cycling greatly increases TBC life in terms of time at temperature. However, the fact that the improvement is not 19-fold (ratio of time at test temperature in the long cycles to that in the short cycles, i.e., 57/3) indicates that life is governed by a combination of a thermal cycling component as well as a time at temperature component. The thermal exposure component may be associated more so with poor oxidation resistant bond coatings or with the ceramic coating when oxidation resistant bond coatings are used. The results are in agreement with the findings reported by McDonald and Hendricks (ref. 13). Their experiments showed that with the poor Ni-16Cr-6Al-0.3Y bond coating, repeatedly subjecting a ceramic coating to high initial heating rates such as those in a Mach 0.3 burner rig was the destructive influence on the coating rather than sustained operation at elevated temperature, whereas a time at temperature component was evident with a better bond coating. Their stress calculations showed that the large thermal stresses, which result from the high initial rates of heat transfer, tend to detach the ceramic coating. These calculated stresses were found to be within the range of measured values of the adhesive/cohesive strength of the ceramic coating.

Figure 7 shows a comparison of the data obtained in the present study with data for the better bond coating reported in reference 13. Here the data are presented in the same manner as in reference 13 and are for the same materials tested under similar conditions. Differences in the two tests are documented in figure 7. As stated above, the trends in the two studies were in agreement. However, the specimens tested in the present study had significantly longer lives (in terms of either time at temperature or number of cycles to failure) in both the short (0.1-hr heating cycles) and long (1.0-hr heating cycles) cyclic tests. It is felt that this is due primarily to the differences in power (11 vs. 20 kW) used in plasma spraying the bond coating. Spraying at 20 kW produced a much better bond coating.

At least a three-fold improvement in life (short or long cycles) was obtained with the best bond coating. This is substantiated by the data presented in figure 8. Since the TBC performance with the Ni-15.8Cr-12.8Al-0.36Y and Ni-14.1Cr-13.4Al-0.10Zr bond coatings are about the same in the present study (fig. 2), the data of figure 8 show that no significant differences were obtained in the two studies for specimens coated using about the same plasma spraying conditions.

The TBC failures observed visually on the specimens tested using the 1.0- and 0.1-hour heating cycles were similar (typical examples, see figs. 3(a) and 3(b)). However, all the specimens tested using the 1.0-hour heating cycles also visually showed a "mud-flat" network after testing, but only on the front surfaces. Figure 9 shows examples of the mud-flat network. As is evident in this figure, the network became more prominent as testing continued. It is felt that the network is a result of microcracking due to a combination of thermal shock cycling, sintering, and subsequent microspalling of the TBC. This is supported by observations noted when several of the tested specimens were examined in a scanning electron microscope (SEM). The topographical features of the TBC after test on two specimens are presented in the SEM photomicrographs of figure 10. Figure 10(a) shows the prominent mud-flat network on the front surface, but only apparent microcracking on the back surface. Figures 9(a) and 10(a) are photographs of the same specimen. Figure 10(b) shows only microcracking of the TBC on both surfaces of a specimen tested in the burner rig using the shorter, 0.1-hour heating cycles. A distinctive mud-flat network has not developed at either location. Note that the degree of microcracking is significantly greater on the back surface of this specimen than on the front. The microcracking of the figure 10(b) specimen is much less extensive than that of the figure 10(a) specimen, because the former was at the maximum exposure temperature for only about 148 hours compared to about 380 hours for the latter.

Figure 11 shows cross-sectional microstructures of a $\text{ZrO}_2\text{-12Y}_2\text{O}_3/\text{Ni-14.1Cr-13.4Al-0.10Zr}$ coated René 41 specimen after 400, 1-hour heating cycles in the 1050° C, Mach 0.3 rig test. Since this specimen after test showed only TBC cracking on the back surface (table II), the ceramic coating is intact on both surfaces. However, both microstructures in figure 11 show a large crack within the ceramic near and along the ceramic/bond coating interface, and numerous fine cracks throughout the ceramic. Note, that the extent of cracking (coarse and fine) is more extensive on the back surface than on the front. As stated above, fine cracks were observed on the external surfaces when examined directly (before sectioning) in the SEM. These observations suggest that the TBC fails by the following mechanism: (1) extension and linking of existing fine cracks throughout the ceramic coating, (2) coarse cracking within the ceramic along the ceramic/bond coating interface, (3) coarse cracking through the ceramic to its outer surface, and then (4) spalling of the ceramic.

There are several phenomena that can generate ceramic coating failure. One is coefficient of thermal expansion mismatch between the ceramic and the coating/substrate components. Another is coating growth as a result of oxidation which can disrupt the ceramic coating. This effect would tend to be driven by time at temperature. However, the bond coating in figures 11 and 4(a) unexpectedly show no significant differences, even though the accumulated times at temperature were vastly different (about 380 and 137 hr, respectively). The bond coating is far from being exhausted of life at the time the ceramic fails, i.e., very little oxidative degradation of the bond coating had occurred in both specimens. Using an energy dispersive X-ray

spectrometer in a SEM, microprobe analysis indicated that the aluminum content of the bond coating was not significantly depleted in either case after TBC failure. Thus, in the present study, TBC life appears to be limited by crack initiation and growth in the ceramic coating induced by a combination of thermal exposure and cyclic thermal stress.

Several phenomena can occur within the ceramic which would have a bearing on coating failure. One is crack growth which would tend to be driven by thermal stress cycles. Another is ceramic sintering or transformations which may play a role in the response of the ceramic to thermal stress. This phenomenon is driven by time at temperature. Based on the results of the present study, as well as those of McDonald and Hendricks (ref. 13) with the better bond coating, both time at temperature and thermal cycling play significant roles in ceramic coating life. Additional research is required to develop a phenomenological description.

CONCLUSIONS

As a result of this study of the effects of bond coating composition, thickness, and plasma spray deposition parameters on the durability of the $\text{ZrO}_2\text{-12Y}_2\text{O}_3$ thermal barrier coating (TBC) in short (0.1 hr) and long (1.0 hr) cycles to 1050°C in a Mach 0.3 burner rig, the following conclusions have been drawn:

1. Of eight bond coatings studied, the best bond coatings for the TBC on a nickel-base alloy were Ni-14.1Cr-13.4Al-0.10Zr, Ni-14.3Cr-14.4Al-0.16Y, and Ni-15.8Cr-12.8Al-0.36Y. The bond coatings were sprayed in air to a thickness of 0.015 cm at 20 kW power using argon - 3.5 v/o hydrogen arc gas. With any one of the three coatings, the life of the 0.038-cm thick TBC was at least 2500, 0.1-hour heating cycles in the accelerated burner rig test.
2. With the best bond coatings, bond coating oxidation life was far from being exhausted at the time the ceramic coating failed. Thus, TBC life appeared to be limited by crack initiation and growth in the ceramic coating induced by a combination of thermal exposure and cyclic thermal stress.
3. Thermal cycling frequency had a more life-limiting influence on the TBC than accumulated time at elevated temperature. The TBC lives in terms of time at temperature were increased by a factor of about 2 to 5 when 1.0-hour rather than 0.1-hour heating cycles were used. Since the effect was not 19-fold, a thermal exposure component was important too and appears to be associated with the ceramic coating when highly oxidation-resistant bond coatings are used. At least a three-fold improvement in cyclic life (short and long cycles) was obtained with the best bond coatings.
4. Oxidation resistance of the bond coatings plays an important role in performance of a TBC. Performance was sensitive to the Cr and Y contents of the NiCrAlY coatings. Low Cr coatings (14 to 18 w/o) were better than high Cr coatings (28 to 39 w/o) at the high bond coating Al levels (10 to 14 w/o) investigated in this study. The optimum Y content appears to be 0.3 w/o consistent with previous results.
5. The TBC life depended on the bond coating thickness and plasma spraying conditions. Increasing the thickness from 0.010 to 0.015 cm improved the life by at least 100 percent. Either decreasing or increasing the spraying power from 20 kW resulted in poorer TBC performance, i.e., about 20 to 90 percent loss in life.

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TABLE I. - CHEMICAL ANALYSES OF PLASMA

SPRAY POWDERS

Element	Content, w/o	
	Thermal barrier oxide	Bond coatings ^a
Al	0.06	11.91 - 19.85 ^b
B	ND	0.018
C	ND	.022
Ca	.24	<.001
Co	ND	.075
Cr	ND	14.06 - 38.58 ^b
Cu	ND	.025
Fe	.16	.079
Hf	2.00	<.01
Mg	.06	<.001
Mn	ND	.041
Mo	ND	.005
Nb	ND	.018
Ni	ND	Major
O ₂	ND	.089
P	ND	.008
S	ND	.004
Si	.23	.12
Ta	ND	.12
Ti	.06	.008
V	ND	.008
W	.01	<.01
Y	9.11	0.11 - 0.69 ^b
Zn	ND	.033
Zr	Major	.10

^aImpurity values reported for the bond coating powders are the maximum values encountered.

^bAluminum, chromium, and yttrium concentrations for the bond coatings are given in the report.

TABLE II. - RESULTS OF CYCLIC MACH 0.3 BURNER RIG OXIDATION AT 1050° C FOR ZrO₂-12Y₂O₃ PLUS BOND COATINGS ON RENÉ 41

Target bond coating thickness, cm	Plasma arc power, kW	Plasma arc gas	Bond coating composition, w/o	Measured bond coating thickness, ^a cm	TBC failure		TBC failure mode	
					Cycles	Approximate hours at maximum temperature ^b	Ceramic top coating	Bond coating
0.1-Hour heating cycles								
0.015 ↓	20 ↓	Ar-3.5 vol % H ₂ ↓	Ni-14.1Cr-13.4Al-0.10Zr Ni-14.1Cr-14.4Al-0.16Y Ni-15.8Cr-12.8Al-0.36Y Ni-17.7Cr-12.2Al-0.11Y Ni-30.7Cr-12.0Al-0.25Y Ni-28.8Cr-19.9Al-0.59Y Ni-30.9Cr-11.1Al-0.48Y Ni-38.6Cr-10.4Al-0.69Y	0.015, 0.015 .015, .015 .014, .014 .015, .017 .014, .015 .015, .014 .014, .017 .014, .015	3788±69, 2713±35 3155±85, 1953±6 3253±72, 2535±74 1351±65, 2160±36 1485±89, 1551±70 668±69, 668±70 452±70, 448±86 446±22, 294±70	189, 136 158, 148 163, 127 68, 108 74, 78 33, 33 23, 22 22, 15	(c), (c) ↓	(d) ↓ (c), (c) (c), --- (c), (c)
0.010	20	Ar-3.5 vol % H ₂	Ni-14.1Cr-13.4Al-0.10Zr	0.011, 0.011	848±67, 1365±80	42, 68	(c), (c)	---, ---
0.015	16	Ar-3.5 vol % H ₂	Ni-14.1Cr-13.4Al-0.10Zr	0.015, 0.015	610±15, 907±88	31, 45	(c), (c)	---, ---
0.015	14	Ar	Ni-14.1Cr-13.4Al-0.10Zr	0.015, 0.015	1208±30, 207±28	60, 14	(c), (c)	---, ---
0.015	24	Ar-3.5 vol % H ₂	Ni-15.8Cr-12.8Al-0.36Y	0.015, 0.015	1311±69, 1951±20	66, 98	(c), (e)	---, ---
1.0-Hour heating cycles								
0.015	20	Ar-3.5 vol % H ₂	Ni-14.1Cr-13.4Al-0.10Zr	0.014, 0.015	770±10, 390±10	732, 380	(c), (e)	---, ---
			Ni-15.8Cr-12.8Al-0.36Y	.015, .015	530±10, 570±10	504, 542	(c), (c)	---, ---

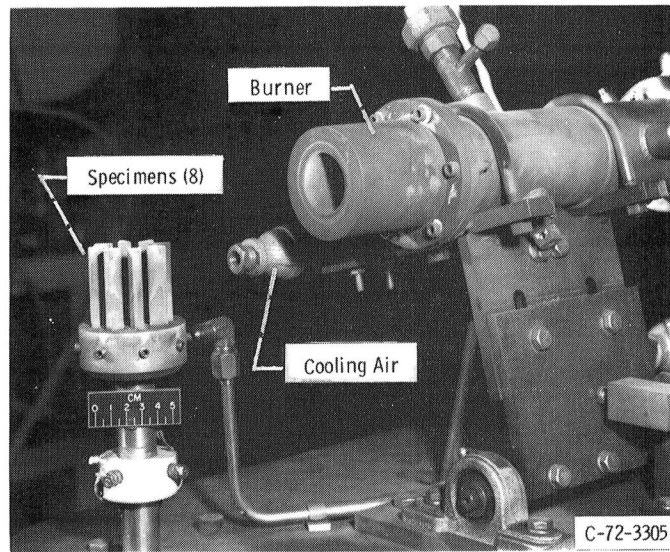
^aMeasured with a vernier caliper average values are listed. The measured TBC thickness values varied from 0.034 to 0.042 cm.

^bAssuming 3 minutes required to heat specimens to test temperature.

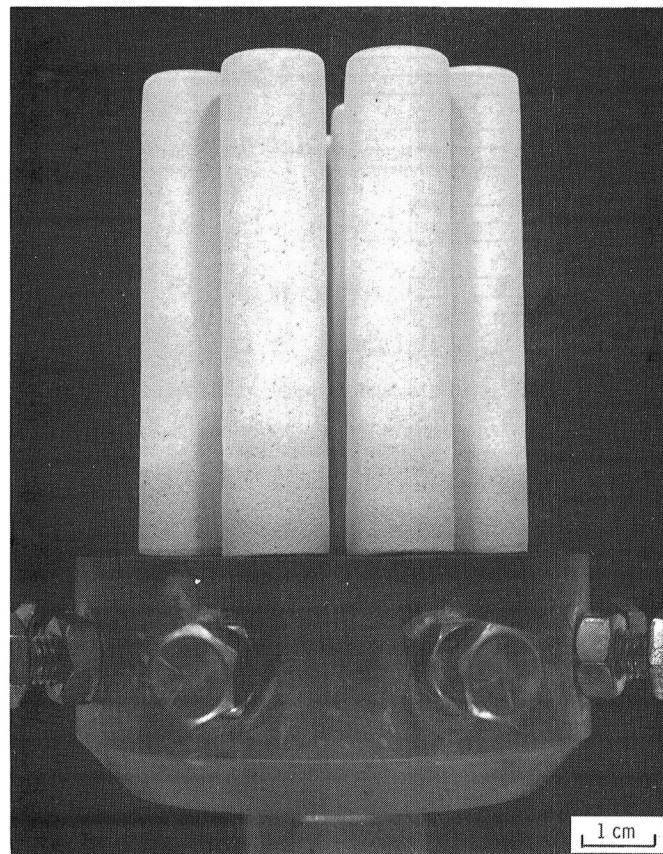
^cCracking followed by spalling.

^dNone visually observed.

^eCracking only.



(a) Overall view.



(b) Specimen holder assembly.

Figure 1. - Mach 0.3 oxidation apparatus.

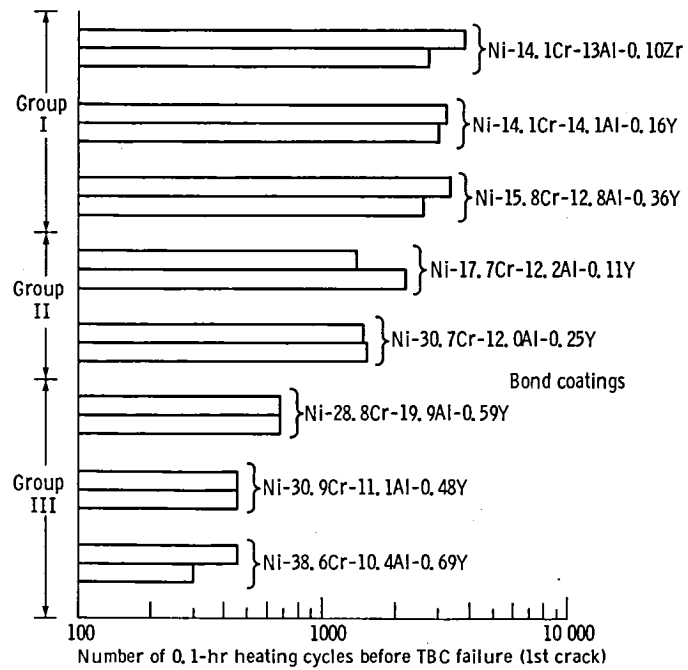
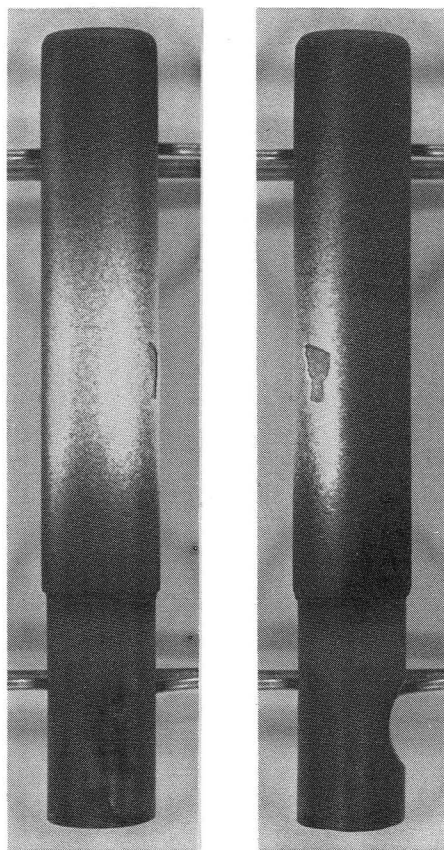
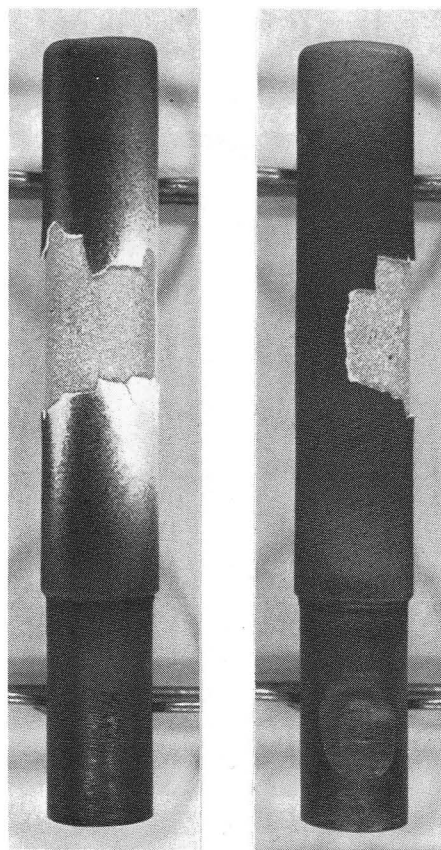


Figure 2. - Performance of TBC systems on solid René 41 pins in Mach 0.3 burner rig oxidation.



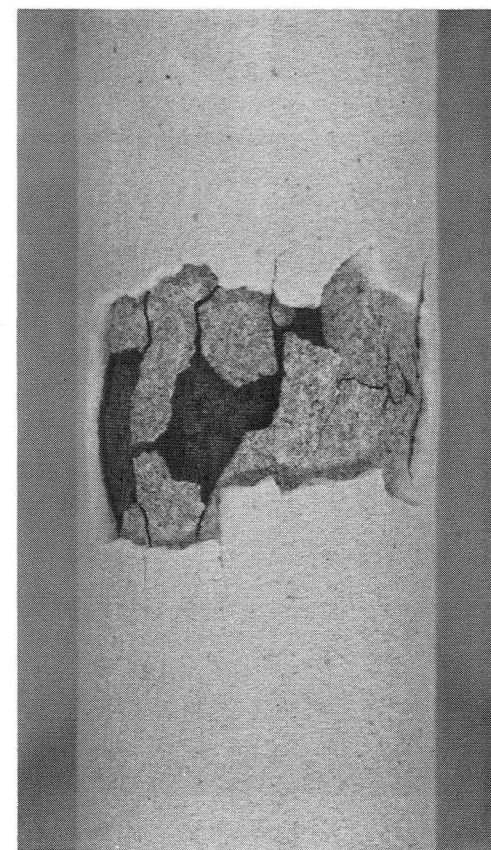
Back surface 1 cm Side surface

(a) 2959 cycles Ni-14, 1Cr-13, 4Al-0, 1Zr bond coating.



Back surface 1 cm Front surface

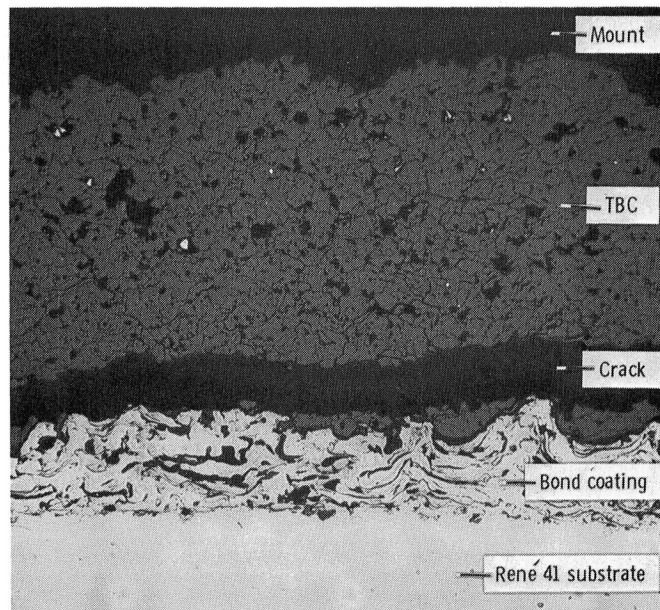
(b) 3240 cycles Ni-14, 1Cr-14, 4Al-0, 16Y bond coating.



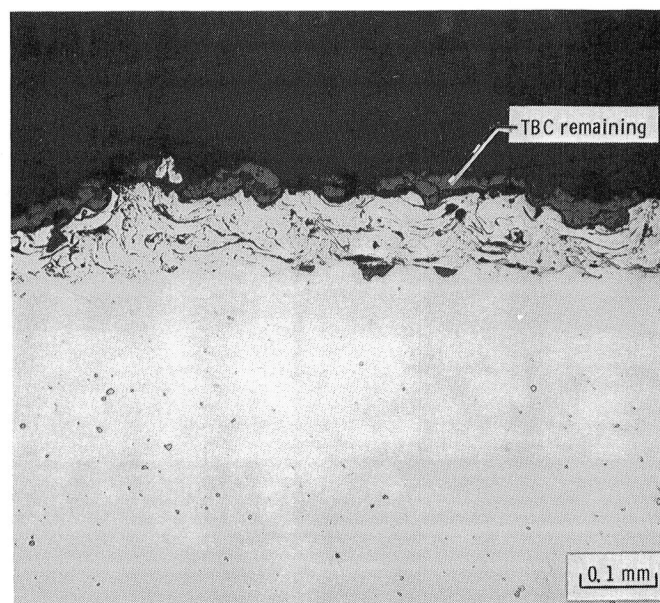
3 mm Back surface

(c) 364 cycles Ni-38, 6Cr-10, 4Al-0, 69Y bond coating.

Figure 3. - Photographs of TBC René 41 solid pins after cyclic oxidation exposure in a Mach 0.3 burner rig. Maximum temperatures at 3.2 cm from top: 1050° C at center line of pin, ~1150° C on back surface. Heating cycles: 0.1 hour.



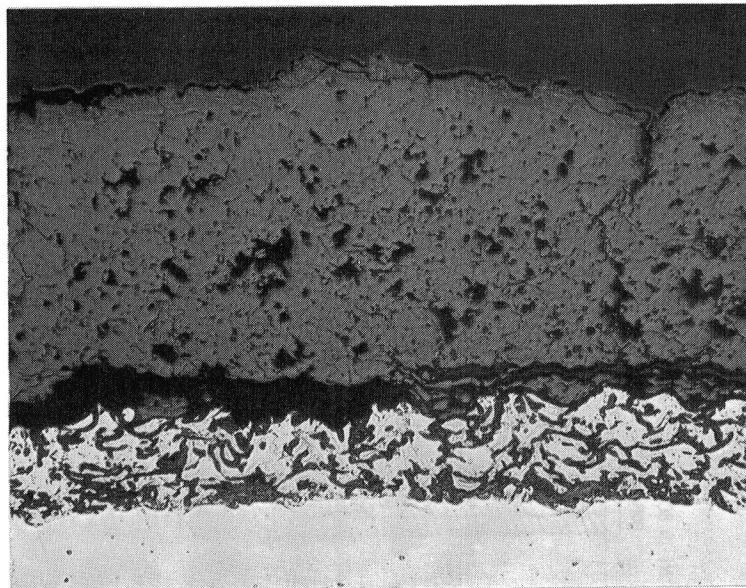
Front surface



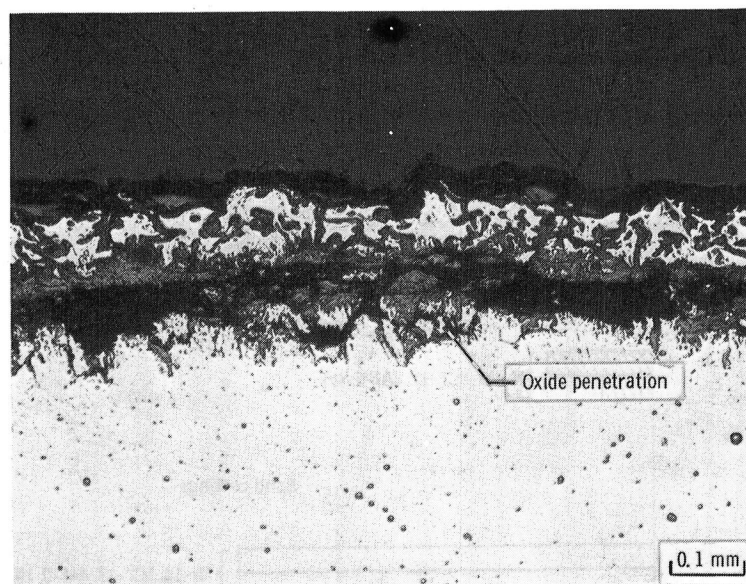
Back surface

(a) $\text{ZrO}_2\text{-}12\text{Y}_2\text{O}_3/\text{Ni-}14, 1\text{Cr-}13, 4\text{Al-}0, 10\text{Zr}$ TBC system after 2748 cycles.

Figure 4. - Photomicrographs of TBC René 41 pins after cyclic oxidation testing in a Mach 0,3 burner rig at 1050°C . Heating cycles: 0,1 hour. Bond coatings applied at 20 kW using argon-3,5 vol hydrogen. TBC: 0,038 cm.



Front surface



Back surface

(b) $\text{ZrO}_2\text{-12Y}_2\text{O}_3/\text{Ni-28, 8Cr-19, 9Al-0.59Y}$ TBC system after 738 cycles.

Figure 4. - Concluded.

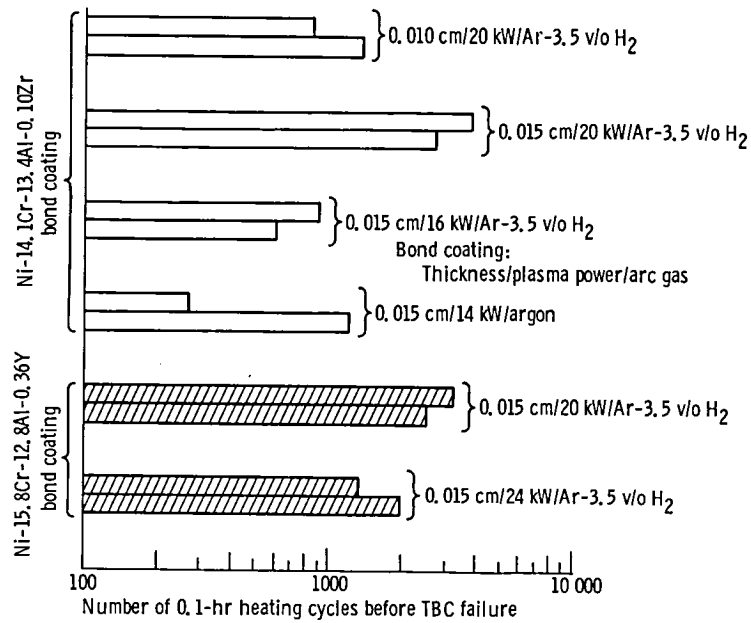


Figure 5. - Effect of bond coating thickness and plasma spraying conditions on performance of $ZrO_2-12Y_2O_3$ TBC systems on solid René 41 pins in Mach 0.3 burner rig oxidation. TBC thickness: 0.038 cm. Maximum exposure temperature at center of pins: $1050^{\circ}C$.

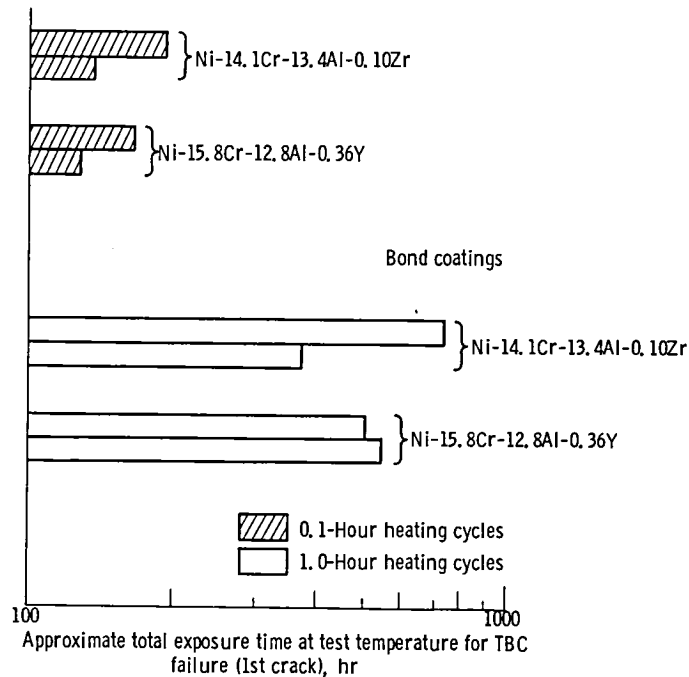


Figure 6. - Effect of cycling frequency on performance of TBC systems on solid René 41 pins in Mach 0.3 burner rig oxidation. Bond coatings: 0.015-cm thick applied at 20 kW using argon - 3.5 v/o hydrogen plasma arc gas. TBC: 0.038-cm thick $ZrO_2-12Y_2O_3$. Maximum temperature at center of substrate: $1050^{\circ}C$.

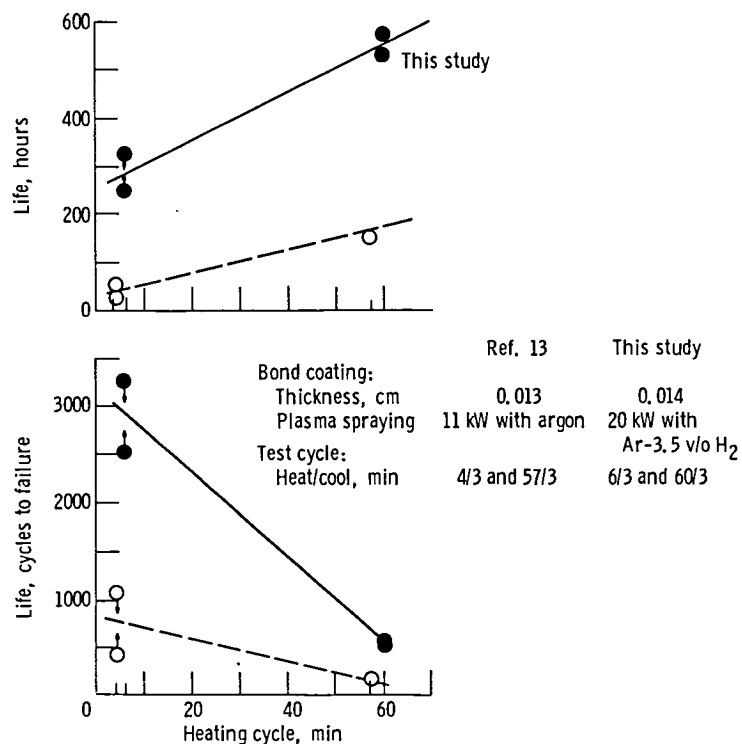


Figure 7. - Effect of cycling frequency on life of TBC $ZrO_2-12Y_2O_3$ on Ni-15.8 Cr-12.8Al-0.36Y on solid Rene 41 pins in a Mach 0.3 burner rig at 1050° C.

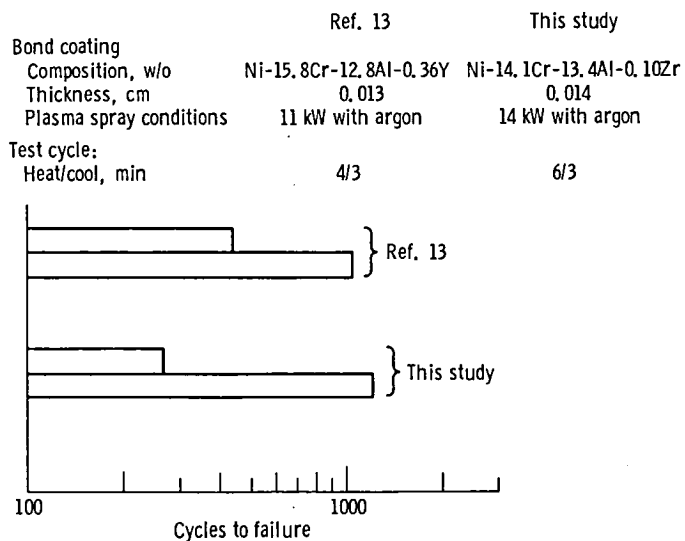
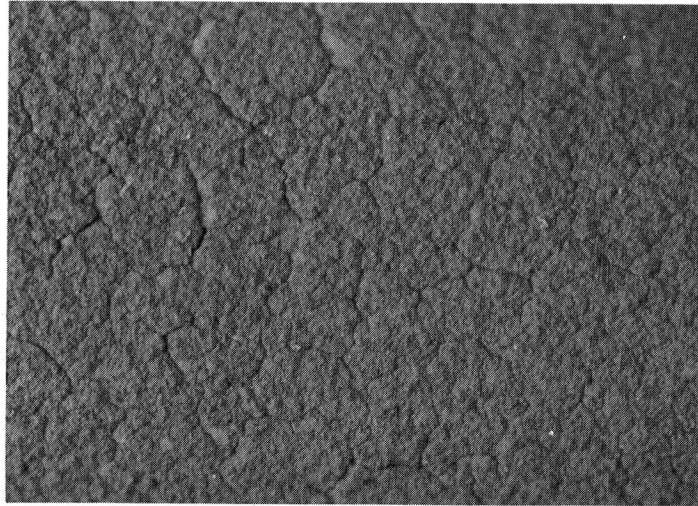
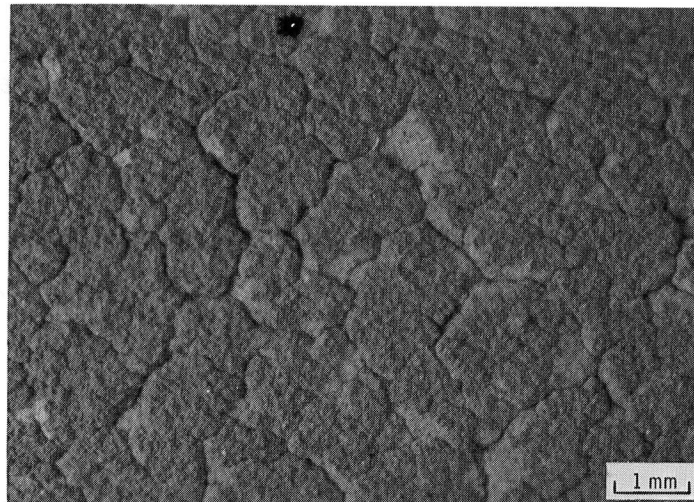


Figure 8. - Comparison of $ZrO_2-12Y_2O_3$ TBC lives obtained in reference 13 and in this study.

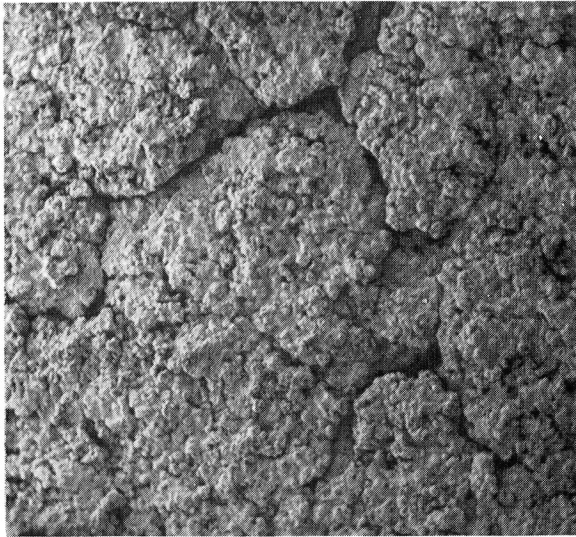


(a) 400 cycles.



(b) 780 cycles.

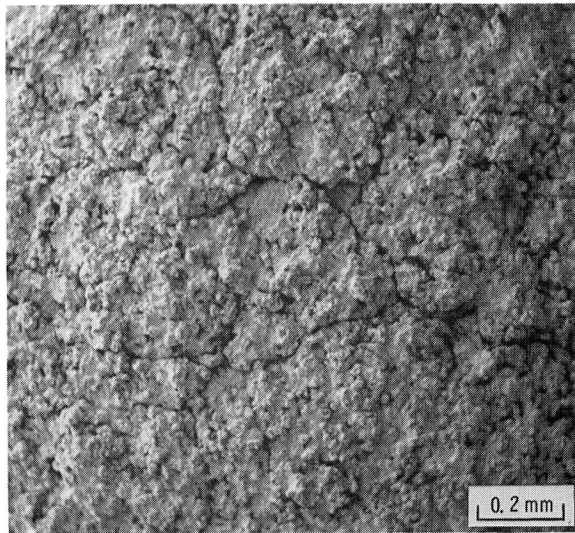
Figure 9. - Photographs of front surfaces of $\text{ZrO}_2\text{-}12\text{Y}_2\text{O}_3\text{/Ni-}14.1\text{Cr-}13.4\text{Al-}0.10\text{Zr}$ coated René 41 pins after cyclic oxidation exposure in a Mach 0.3 burner rig at 1050°C . Heating cycles: 1 hour.



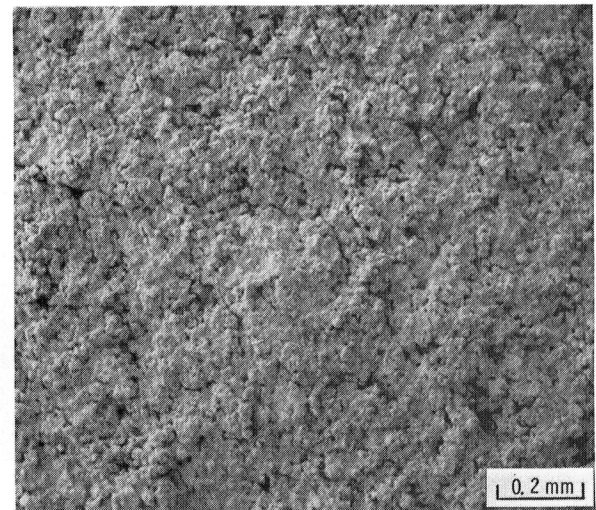
(a-1) Front surface.



(b-1) Front surface.



(a-2) Back surface.



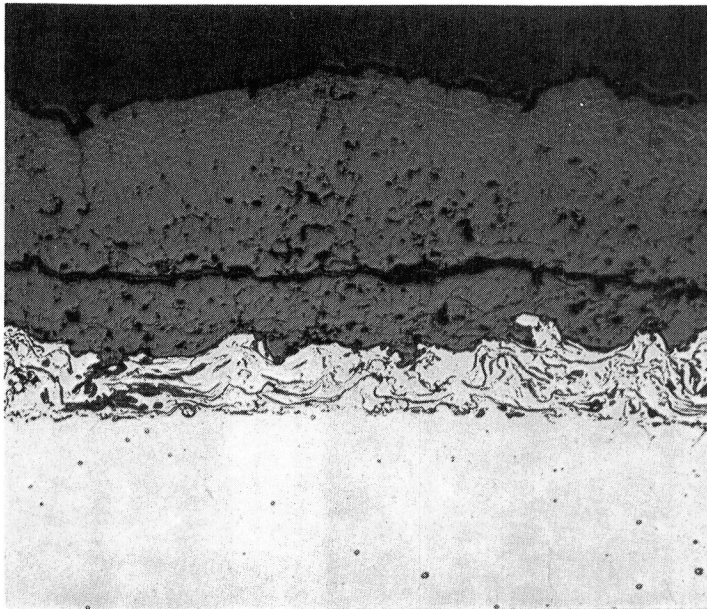
(b-2) Back surface.

(a) 400, 1-hour cycles (380 hr at maximum exposure temperature); $\text{ZrO}_2\text{-}12\text{Y}_2\text{O}_3\text{/Ni-}14, 1\text{Cr-}13, 4\text{Al-}0, 1\text{Zr}$ coating.

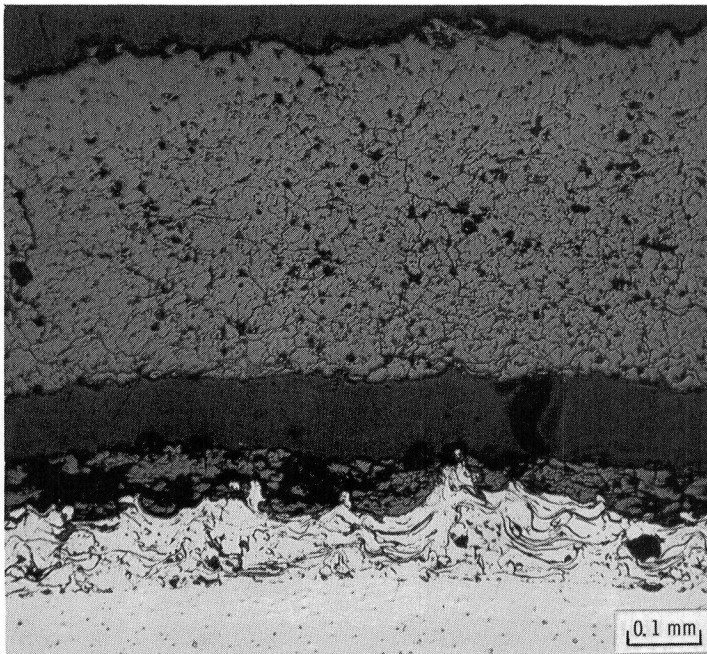
(b) 2959, 0.1-hour cycles (~148 hr at maximum exposure temperature); $\text{ZrO}_2\text{-}12\text{Y}_2\text{O}_3\text{/Ni-}14, 1\text{Cr-}14, 4\text{Al-}0, 16\text{Y}$ coating.

Figure 10. - Scanning electron photomicrographs of TBC systems on René 41 pins after cyclic oxidation exposure in a Mach 0.3 burner rig at 1050°C .

Figure 10. - Concluded.



Front surface



Back surface

Figure 11. - Photomicrographs of $\text{ZrO}_2\text{-}12\text{Y}_2\text{O}_3\text{-}14\text{Ni-}13.4\text{Al-}0.12\text{r}$ coated René 41 pins after 400, 1-hour heating cycles in a Mach 0.3 burner rig at 1050°C . Bond coating: 0.015 cm thick applied at 20 kW using argon-3.5 v/o hydrogen. TBC: 0.038 cm thick.

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16. Abstract Eight plasma-sprayed bond coatings were evaluated for their potential use with $ZrO_2-Y_2O_3$ thermal barrier coatings (TBCs) which are being developed for coal-derived-fuel-fired gas turbines. Longer TBC lives in cyclic burner rig oxidation to $1050^{\circ}C$ were achieved with the more oxidation-resistant bond coatings. These were Ni-14.1Cr-13.4Al-0.10Ar, Ni-14.1Cr-14.4Al-0.16Y, and Ni-15.8Cr-12.8Al-0.36Y on René 41. The TBC systems performed best when 0.015-cm thick bond coatings were employed that were sprayed at 20 kW using argon - 3.5 v/o hydrogen. Cycling had a more life limiting influence on the TBC than accumulated time at $1050^{\circ}C$.					
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